



(19) JAPANESE PATENT OFFICE (JP)

(12) Publication of Unexamined Patent Application (KOKAI) (A)

(11) Japanese Patent Application Kokai Number: **H8-5528**

(43) Kokai Publication Date: January 12, 1996

(51) Int. Cl. <sup>6</sup>	Identification Symbol	JPO File No.	F1	Technical Indication
G 01 N 1/28				
H 01 J 37/20	Z			
37/28	Z			
37/30	Z	9172-5E		
			G 01 N 1/28	F
Request for Examination: Not requested		Number of Claims: 4	OL	(10 pages total)
				Continue to the last page

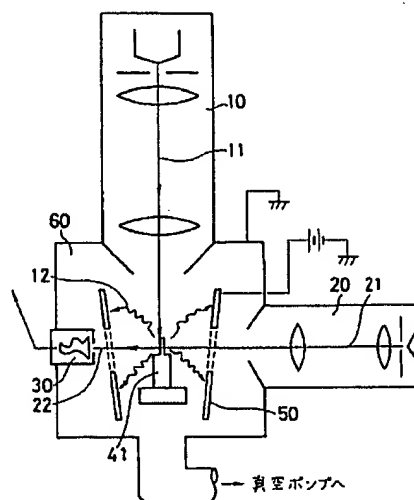
(21) Application Number: H6-141852	(71) Applicant: 000005049 Sharp Corporation 22-22 Nagaike-cho, Abeno-ku, Osaka-shi, Osaka
(22) Filing Date: June 23, 1994	(72) Creator: Fumitoshi Yasuo c/o Sharp Corporation 22-22 Nagaike-cho, Abeno-ku, Osaka-shi, Osaka
	(74) Agent: Yoshio Kawaguchi, Patent Attorney (and one other)

(54) [Title of the Invention] **FOCUSED ION BEAM APPARATUS FOR PRODUCING CROSS-SECTIONAL SAMPLE FOR TRANSMISSION ELECTRON MICROSCOPE AND METHOD FOR PRODUCING CROSS-SECTIONAL SAMPLE FOR TRANSMISSION ELECTRON MICROSCOPE**

(57) [Abstract]

[Object] [The object of the present invention is] to provide a focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope which makes it possible to automatically detect the working endpoint at the optimal sample thickness by quantitatively monitoring the thickness of the worked surface of the transmission electron microscope sample, and which also allows easy judgement of the uniformity of the thickness of the worked part during working, and a method for producing such a sample.

[Constitution] [The apparatus of the present invention] comprises a working chamber 60 in which a cross-sectional sample 41 for a transmission electron microscope is disposed, an ion gun 10 which emits an ion beam 11 onto the sample 41 disposed in the working chamber 60, an electron gun 20 which irradiates the worked portion of the sample 41 with an electron beam 21 at an angle of approximately 90 degrees with respect to the ion beam 11 emitted by the ion gun 10, a transmission electron detector 30 which is disposed facing the above-mentioned electron gun 20, and which receives the electron beam that is transmitted through the sample 41 and detects the amount of current of the transmitted electron beam, and a low-voltage electrode 50 which is positioned so that this electrode surrounds the sample 41 in the vicinity of the position where the above-mentioned sample 41 is fixed, and which absorbs the secondary electrons 12 generated by the ion beam 11 and electron beam 12.



[Key:] To vacuum pump

**[Claims]**

**[Claim 1]** A focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope, comprising ion gun means which emit an ion beam that is used to produce a cross-sectional sample for a transmission electron microscope, electron gun means which irradiate the worked portion of the above-mentioned cross-sectional sample for a transmission electron microscope with an electron beam at an angle of approximately 60 to 90 degrees with respect to the ion beam emitted by the above-mentioned ion gun means, and detection means which are disposed facing the above-mentioned electron gun means, and which receive the electron beam that is transmitted through the above-mentioned cross-sectional sample for a transmission electron microscope, and detect the amount of current of the transmitted electron beam.

**[Claim 2]** The focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope according to Claim 1, which comprises electrode means that absorb the secondary electrons generated by the irradiation with the ion beam and electron beam in the vicinity of the position where the above-mentioned cross-sectional sample for a transmission electron microscope is fixed.

**[Claim 3]** A method for producing a cross-sectional sample for a transmission electron microscope comprising the steps of cutting a sample requiring cross-sectional observation by a transmission electron microscope to a thickness that allows mounting in such a transmission electron microscope, making the observation region even thinner by means of an ion beam, irradiating the worked part of the above-mentioned sample with an electron beam while [the sample is] being made thinner by means of the above-mentioned ion beam, detecting the amount of current of the electron beam that is transmitted through the above-mentioned sample, and evaluating the uniformity of the thickness of the worked part of the above-mentioned sample on the basis of the above-mentioned detected current amount by scanning this worked part with the above-mentioned electron beam.

**[Claim 4]** A method for producing a cross-sectional sample for a transmission electron microscope comprising the steps of cutting a sample requiring cross-sectional observation by a transmission electron microscope to a thickness that allows mounting in such a transmission electron microscope, making the observation region even thinner by means of an ion beam, irradiating the worked part of the above-mentioned sample with an electron beam while [the sample is] being made thinner by means of the above-mentioned ion beam, detecting the amount of current of the electron beam that is transmitted through the above-mentioned sample, and

detecting the working endpoint of the above-mentioned sample on the basis of the above-mentioned detected current amount.

[Detailed Description of the Invention]

[0001]

[Field of Industrial Utilization] The present invention relates to a focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope and a method for producing a cross-sectional sample for a transmission electron microscope, and more particularly relates to a focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope and a method for producing a cross-sectional sample for a transmission electron microscope which produce a cross-sectional sample (for a transmission electron microscope) of a specified microscopic part such as the location of a defect on an LSI chip.

[0002]

[Prior Art] Recently, as LSI devices have become finer and films of LSI materials have become thinner, the observation and evaluation of fine structures that determine LSI device performance have become extremely important. In particular, extremely thin films with thicknesses of a few nanometers have been used as gate insulating films of transistors, and a high spatial resolution with a few tenths of a nanometer or less is considered necessary for the observation and evaluation of such fine structures. Furthermore, the evaluation of crystal defects, which are a cause of leaks in the fine transistors of LSI devices, and which lead to various types of defects, is also extremely important for improving the performance and improving the yield of LSIs. A transmission electron microscope (TEM) is the only evaluation device that can achieve such objects.

[0003] A transmission electron microscope has the highest spatial resolution among high-resolution observation and evaluation devices, i.e., approximately 0.2 nm, and is the only means of observing and evaluating LSI gate insulating films, etc., that are formed as ultra-thin films. Furthermore, a transmission electron microscope is also the only device that can directly observe crystal defects with a high spatial resolution. Moreover, a transmission electron microscope is capable not only of observation, but also of elemental analysis at a spatial resolution of approximately 1 nm in combination with an X-ray microanalyzer (EPMA), etc., and has a spatial resolution that is approximately 1/20 that of the Auger electron spectroscopic analysis method (AES), which has the highest spatial resolution among other analysis methods. Accordingly, such a transmission electron microscope has the status of an extremely useful analysis tool in the analysis of LSI devices of progressively greater fineness.

[0004] A projected image of an electron beam that is transmitted through the sample is used in observation and evaluation performed by means of a transmission electron microscope.

Accordingly, samples used in a transmission electron microscope must be worked to a thickness that allows the transmission of such an electron beam. In concrete terms, it is necessary to form [the sample] into a thin film with a thickness of approximately 500 nm or less, and in order to perform high-resolution observations for the purpose of evaluating the crystal structure, etc., in particular, it is necessary to form the sample into a thin film with a thickness of approximately 100 nm or less.

[0005] Generally, the preparation of LSI samples for use in a transmission electron microscope is accomplished by performing the final thin film formation by means of an ion beam after the samples have been thinned by mechanical means. However, this technique is used in cases where thin films formed over a broad range, or arbitrary locations in an LSI pattern in which the same shape is repeated, are the object [of observation]. In cases where specified locations on finely worked LSIs, e.g., defective transistors or open contacts, are evaluated, there is a danger that the observation and evaluation location will be lost if the position of the worked part of thin film formation shifts. Accordingly, in the preparation of samples for a transmission electron microscope, it is necessary to form a specified location into a thin film with a positional precision of 1  $\mu\text{m}$  or better. This cannot be accomplished using simple mechanical polishing and ion beam working. Accordingly, several sample working methods have been proposed.

[0006] Below, a method in which a transmission electron microscope sample used for transmission electron microscope observation and analysis of the cross sections of specified microscopic parts such as the locations of defects on LSI chips is worked by mechanical polishing will be described using Figures 4a through 4g.

[0007] (1-1) As is shown in Figure 4a, marking 43 is performed by forming holes around a specified microscopic part 42 for which transmission electron microscope observation is desired by means of a laser marker or focused ion beam apparatus, etc., equipped with a microscope. Furthermore, in order to protect the specified microscopic part 42 from the thermal effects of laser or ion beam irradiation or contamination by flying debris from the hole formation process used for marking 43, it is advisable to perform the marking in positions that are separated from the specified microscopic part 42 by a distance of approximately 20  $\mu\text{m}$  or greater. From the standpoint of positional confirmation in subsequent working, it is desirable that the size and depth of the marking be as large as possible; on the other hand, from the standpoint of the need to suppress heat and flying debris during marking, it would appear desirable that the size [of the marking] be approximately 5  $\mu\text{m}$  or less, and that the depth [of the marking] be approximately 1

to 5  $\mu\text{m}$ . In cases where it is necessary to use a low-power microscope such as a stereoscopic microscope in the working of the sample, it is advisable to add marking with a size of approximately 10  $\mu\text{m}$  to the above-mentioned marking in a position that is separated from the specified microscopic part 42 by a distance of 40  $\mu\text{m}$  or greater.

[0008] (1-2) A glass 44 is pasted to the surface of the sample 41 in order to protect the surface.

[0009] (1-3) Using the marking as a reference, the area around the specified microscopic region for which observation or analysis is desired is cut by means of the high-speed outer circumferential rotary blade 61 of a dicing machine to a size of approximately 1.5 mm square or less, which allows introduction [of the sample] into a transmission electron microscope. In this case, as is shown in Figures 4b and 4c, [it is desirable to] select surfaces that are parallel to the sectional surface for which observation or analysis of the transmission electron microscope sample 41 is desired, and surfaces that are perpendicular to this surface, as the cut surfaces. In regard to the cutting width in the direction perpendicular to the sectional surface for which observation/analysis of the sample 41 is desired, a narrower width makes it possible to shorten the time required for the subsequent polishing; accordingly, cutting is performed at a narrow width within a range that causes no destruction of the specified microscopic part 42 (for which observation or analysis is desired) during cutting, e.g., 100 to 200  $\mu\text{m}$ .

[0010] (1-4) As is shown in Figures 4d and 4e, the two cut surfaces parallel to the sectional surface for which observation or analysis of the sample 41 is desired are mechanically polished by means of a polishing tool 70 and a rotary polishing platen 71. In this case, using the marking as a reference, one side surface is polished until a distance of approximately 10  $\mu\text{m}$  is reached with respect to the specified microscopic part 42 for which observation/analysis is desired. The other side surface that is opposite this first side surface of the sample 41 is polished until a distance of approximately 70  $\mu\text{m}$  is reached with respect to the specified microscopic part 42. As a result, the width of the sample 41, which is the interval between the polished surfaces, is approximately 80  $\mu\text{m}$ . Furthermore, polishing grains with a size of approximately 5 to 15  $\mu\text{m}$ , which have a relatively rapid polishing speed, are used for the polishing up to this point. The polished surface on the first side surface of the sample 41, which is close to the specified microscopic part 42, is subjected to mirror finishing using even finer polishing grains with a size of 1  $\mu\text{m}$  or less in this stage.

[0011] (1-5) As is shown in Figure 4f, the sample 41 is fastened to the surface of a rotating stage 73 with the polished surface that has not been subjected to mirror finishing, i.e., the second side surface of the sample 41 which is distant from the specified microscopic part 42, facing upward, and is subjected to dimple grinder polishing centered on the portion for which

observation/analysis is desired by means of a rotary polishing disk 72. In this dimple grinder polishing, polishing is first performed using a polishing material with a size of 5 to 10  $\mu\text{m}$  until the thickness in the vicinity of the portion for which observation/analysis is desired reaches 20 to 30  $\mu\text{m}$ . Then, mirror finishing of the portion for which observation/analysis is desired is performed using polishing grains with a size of 1  $\mu\text{m}$  or less.

[0012] (1-6) As is shown in Figure 4g, [the sample 41] is pasted to a transmission electron microscope mesh 80 with the portion of the sample 41 for which analysis/observation is desired at the center.

[0013] (1-7) Ion milling is performed on both sides using an ion milling device so that a thickness of 500 nm or less is obtained.

[0014] (1-8) Observation and analysis of the sample 41 are performed by means of a transmission electron microscope.

[0015] Next, a transmission electron microscope sample working method using a focused ion beam apparatus disclosed in Japanese Patent Application Kokai No. H2-132345 and Japanese Patent Application Kokai No. H5-180739 will be described with reference to Figures 5a through 5g.

[0016] (2-1) As is shown in Figures 5a through 5c, marking is performed on the sample, and cutting of the sample 41 is performed by the same methods as in the above-mentioned (1-1) and (1-3). If necessary, the region of the sample for which observation/analysis is desired is ground even thinner by means of the high-speed outer circumferential rotary blade 61 of a dicing machine as shown in Figure 5d.

[0017] (2-2) As is shown in Figures 5e and 5f, the area in the vicinity of the specified microscopic part 42 for which observation/analysis is desired is irradiated from the direction of the sample surface with a focused ion beam 11 by means of a focused ion beam apparatus. In this case, as is shown in Figure 5g, the focused ion beam 11 is raster-scanned over rectangular regions 81 and 82 that have one side parallel to the sectional surface for which observation/analysis is desired, and these regions are etched by sputtering. The raster scanning regions are gradually caused to approach the sectional surface for which observation/analysis is desired while the beam current and beam diameter, etc., of the focused ion beam 11 are appropriately selected, so that sectional surface working is performed as shown in Figure 5f. The specified microscopic part 42 for which observation/analysis is desired is formed into a thin film by performing this working from both sides of this microscopic part, thus producing a transmission electron microscope sample.

[0018] Furthermore, as is shown in Figure 6 (c), if the focused ion beam 11 has an inverted circular conical shape, and beam irradiation is performed perpendicular to the sample surface, a perpendicular sectional surface cannot be obtained. Accordingly, a perpendicular sectional surface is obtained by inclining the sample 41 by a specified angle of  $\theta$ . Since this angle  $\theta$  varies according to the focused ion beam apparatus and working conditions, conditions must be determined in advance, and working is generally performed at an inclination of approximately 3 to 5 degrees. Furthermore, during actual working, the working process is intermittently interrupted, the worked shape is evaluated through the observation of a secondary ion image or secondary electron image obtained by means of the focused ion beam, observation with the sample transferred to a scanning electron microscope, or observation of a secondary electron image produced by irradiation with an electron beam inside the apparatus (in the case of a focused ion beam apparatus which has an electron beam irradiation function), etc., and in cases where there is a problem, adjustment of the focused ion beam, alteration of the conditions, or adjustment of the angle of the sample, is appropriately performed.

[0019] (2-3) With the portion of the sample 41 for which analysis/observation is desired placed at the center, [the sample] is pasted to a transmission electron microscope mesh 80 as shown in Figure 5g.

[0020] (2-4) The sample 41 is observed and analyzed by means of the transmission electron microscope.

[0021] The endpoint of focused ion beam working [may be] determined by the following methods:

[0022] (1) The shape of the worked part is observed using a secondary ion image or secondary electron image, etc., obtained by ion beam irradiation, and the working endpoint is determined by judging the thickness of the worked part from the observed image. Furthermore, the image resolution is several tens of nanometers.

[0023] (2) Focused ion beam working and scanning electron microscopic observation are alternately performed, and the working endpoint is determined by judging the thickness of the worked part from the image of the worked part observed by means of the scanning electron microscope. Alternatively, focused ion beam working and scanning electron microscopic observation are alternately performed, and the degree of completion of the sample is judged from the resolution of the transmission electron microscopic observation.

[0024] (3) As is disclosed in Japanese Patent Application Kokai No. H4-76437, in a focused ion beam apparatus which is equipped with an electron gun separately from the ion gun, or in a

focused ion beam apparatus which can perform electron beam irradiation using the ion gun, focused ion beam working and observation by means of an electron beam are alternately performed within the focused ion beam apparatus, and the working endpoint is determined by judging the thickness of the worked part from the observed image.

[0025]

[Problems that the Invention is to Solve] In the case of conventional methods for working a transmission electron microscope sample by means of mechanical polishing, the precision of the working position with respect to the microscopic part for which observation and evaluation are desired is a few microns in the mechanical polishing stage. Accordingly, the precision of 1  $\mu\text{m}$  or better that is required in working for the purpose of observing the locations of defects in LSIs cannot be obtained.

[0026] In the case of conventional methods for working a transmission electron microscope sample by means of a focused ion beam apparatus, when the worked shape is evaluated through the observation of a secondary ion image or secondary electron image created by a focused ion beam, or when the working endpoint is judged by evaluating the thickness of the worked surface through the observation of a secondary ion image or secondary electron image created by a focused ion beam, the target thickness of working is several tens to several hundreds of nanometers. However, the beam system [sic]<sup>\*</sup> of the focused ion beam is at least approximately 100 nm, and since the resolution of the secondary ion image or secondary electron image that is obtained also depends on the ion beam diameter, it is difficult to judge the accurate thickness on the screen, so that the success rate of sample preparation drops. Since observation and working by means of the focused ion beam are alternately performed, there is a danger that working will be performed beyond the working endpoint. In order to perform sectional working with a focused ion beam having an inverted circular conical shape, the sample is inclined and worked as shown in Figure 6 (c); however, because of variation in the adjustment of the focused ion beam, etc., the sectional surface that is worked deviates from a plane perpendicular to the surface [of the sample] with each working. For example, in a case where the angle of inclination of both worked surfaces is 2 degrees, the thickness at a position located at a depth of 3  $\mu\text{m}$  shows a deviation of 100 nm with respect to the width of various parts of the outermost surface. Under such conditions, in cases where the width of the worked part reaches the target of 100 nm at the outermost surface 9 [sic]<sup>†</sup>, a width of 200 nm or 0 nm is obtained at a depth of 3  $\mu\text{m}$ , depending

---

<sup>\*</sup> Translator's note: apparent word processing error in the original for "beam diameter"; the terms "system" and "diameter" are homophonous in Japanese.

<sup>†</sup> Translator's note: Nothing in the figures is designated with "9."

on the direction of the inclination, so that a hole is formed. A depth of 3  $\mu\text{m}$  corresponds to the thickness of an LSI device structure from the surface. Furthermore, in cases where the width of the worked part is 200 nm, high-resolution observation such as lattice image observation is difficult. Such an error in the angle of the worked surface with respect to the vertical direction is impossible to evaluate by observation at the observation resolution of the focused ion beam, especially from above, so that the respective thicknesses of the parts for which observation and evaluation are desired cannot be accurately evaluated; accordingly, the success rate of transmission electron microscope sample preparation drops.

[0027] In cases where the worked shape or working endpoint obtained by a focused ion beam is judged using a scanning electron microscope, or in cases where this judgment is made based on the observed image by means of a transmission electron microscope, focused ion beam working and electron microscopic observation are alternately performed; accordingly, time is required for switching of the sample, etc., so that the working time is prolonged. While the working time is generally 3 to 5 hours, if observation is added, a minimum time of approximately 1 hour per observation is required for sample replacement, observation and focused ion beam readjustment, so that even if observation that is performed only two or three times is added, the required time from start to finish of the focused ion beam working is increased by a factor of 1.5 to 2. In cases where focused ion beam working and electron microscopic observation are alternately performed, error in the working direction is generated by the replacement of the sample as shown in Figures 6 (a) and 6 (b) when working is again performed using the focused ion beam. As a result, the thickness of the observed part becomes non-uniform, so that good observation is difficult. In an electron microscope, the observation resolution is a few nanometers or better, so that the thickness of the surface of the observed part can be evaluated more accurately than in the case of an observation method using an ion beam. However, it is difficult to observe and evaluate the worked shape, e.g., to evaluate the angular error of the worked surface with respect to the vertical direction by observation from above, so that the accurate film thickness of the part for which observation is desired cannot be evaluated. When focused ion beam working is again performed following observation, the focused ion beam must be readjusted, and the conditions vary, so that feedback from the evaluation results is also impossible.

[0028] In cases where the judgement of the endpoint of focused ion beam working is accomplished by the observation of a secondary electron image, etc., obtained by means of an electron beam in a focused ion beam apparatus equipped with an electron beam irradiation function, secondary electron image observation by means of an electron beam cannot be performed during focused ion beam working because of the secondary electrons that are generated by ion beam irradiation even in cases where the apparatus has an electron gun that is

separate from the ion gun, to say nothing of cases where the ion gun is also used as an electron gun. Accordingly, working and observation cannot be performed at the same time, and there is a danger that working will be performed beyond the working endpoint. Since the worked part is observed from above, the inclination of the worked surface with respect to the vertical direction cannot be accurately evaluated.

[0029] The present invention was devised in order to eliminate the above-mentioned problems. The object of the present invention is to provide a focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope and a method for producing a cross-sectional sample for a transmission electron microscope which make it possible to automatically detect the optimal sample thickness by quantitatively monitoring the thickness of the worked surface of the transmission electron microscope sample, and which also allow easy judgement of the uniformity of the thickness of the worked part during working.

[0030]

[Means for Solving the Problems] In the present invention, the above-mentioned object is achieved by the focused ion beam apparatus of Claim 1 for producing a cross-sectional sample for a transmission electron microscope, which comprises ion gun means which emit an ion beam that is used to produce a cross-sectional sample for a transmission electron microscope, electron gun means which irradiate the worked portion of the above-mentioned cross-sectional sample for a transmission electron microscope with an electron beam at an angle of approximately 60 to 90 degrees with respect to the ion beam emitted by the above-mentioned ion gun means, and detection means which are disposed facing the above-mentioned electron gun means, and which receive the electron beam that is transmitted through the above-mentioned cross-sectional sample for a transmission electron microscope, and detect the amount of current of the transmitted electron beam.

[0031] In the present invention, the above-mentioned object is achieved by the focused ion beam apparatus of Claim 2 for producing a cross-sectional sample for a transmission electron microscope, which comprises electrode means that absorb the secondary electrons generated by the irradiation with the ion beam and electron beam in the vicinity of the position where the above-mentioned cross-sectional sample for a transmission electron microscope is fixed.

[0032] In the present invention, the above-mentioned object is achieved by the method of Claim 3 for producing a cross-sectional sample for a transmission electron microscope, which comprises the steps of cutting a sample requiring cross-sectional observation by a transmission electron microscope to a thickness that allows mounting in such a transmission electron

microscope, making the observation region even thinner by means of a focused ion beam, irradiating the worked part of the sample with an electron beam while [the sample is] being made thinner by means of the focused ion beam, detecting the amount of current of the electron beam that is transmitted through the sample, and evaluating the uniformity of the thickness of the worked part of the sample on the basis of the above-mentioned detected current amount by scanning this worked part with the electron beam.

[0033] In the present invention, the above-mentioned object is achieved by the method of Claim 4 for producing a cross-sectional sample for a transmission electron microscope, which comprises the steps of cutting a sample requiring cross-sectional observation by a transmission electron microscope to a thickness that allows mounting in such a transmission electron microscope, making the observation region even thinner by means of a focused ion beam, irradiating the worked part of the sample with an electron beam while [the sample is] being made thinner by means of the focused ion beam, detecting the amount of current of the electron beam that is transmitted through the sample, and detecting the working endpoint of the sample on the basis of the above-mentioned detected current amount.

[0034]

[Operation] In the focused ion beam apparatus of Claim 1 for producing a cross-sectional sample for a transmission electron microscope, the sample surface is irradiated with a focused ion beam by the ion gun means at an arbitrary acceleration voltage, beam current and beam diameter, and an arbitrary region on the sample surface is raster-scanned. During ion beam working, the sample is irradiated in an arbitrary position on the worked surface with an electron beam at an angle of approximately 60 degrees to 90 degrees with respect to the ion beam, and at an arbitrary acceleration voltage, beam current and beam diameter, by the electron gun means. Furthermore, the electron beam that passes through the sample is received by the detection means, the amount of current of the transmitted electron beam is detected by the detection means, and working by means of the focused ion beam is ended at a stage in which the current value of the transmitted beam reaches a preset value.

[0035] In the focused ion beam apparatus of Claim 2 for producing a cross-sectional sample for a transmission electron microscope, electrons such as the secondary electrons created by the focused ion beam that irradiates the sample are absorbed by electrode means to which an arbitrary positive voltage is applied, so that [such electrons] do not reach the detection means and interfere with the detection of the amount of the transmission electron beam current.

[0036] In the method of Claim 3 for producing a cross-sectional sample for a transmission electron microscope, when a cross-sectional sample for a transmission electron microscope is to be prepared, the sample requiring cross-sectional observation by means of a transmission electron microscope is cut to a thickness that allows mounting in the transmission electron microscope, the observation region is further thinned by means of the focused ion beam, the worked part is irradiated with an electron beam while being formed into a thin film by means of the focused ion beam, the amount of current of the transmitted beam is detected, the uniformity of the thickness of the worked part of the above-mentioned sample is evaluated on the basis of the detected current value, and a cross-sectional sample for a transmission electron microscope which has a uniform thickness is prepared on the basis of this evaluation.

[0037] In the method of Claim 4 for producing a cross-sectional sample for a transmission electron microscope, when a cross-sectional sample for a transmission electron microscope is to be prepared, such a cross-sectional sample for a transmission electron microscope is prepared by a process in which the sample requiring cross-sectional observation by means of a transmission electron microscope is cut to a thickness that allows mounting in the transmission electron microscope, the observation region is further thinned by means of the focused ion beam, the worked part is irradiated with an electron beam while being formed into a thin film by means of the focused ion beam, the electron beam passing through the sample is monitored, and the working endpoint is detected.

[0038]

[Embodiments] Below, an embodiment of the focused ion beam apparatus of Claim 1 for producing a cross-sectional sample for a transmission electron microscope will be described with reference to Figure 1. The object of the present embodiment is to provide a focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope which can automatically detect the optimal sample thickness by quantitatively monitoring the thickness of the worked surface of the transmission electron microscope sample, and which can easily judge the uniformity of the thickness of the worked part during working.

[0039] The present embodiment comprises a working chamber 60 in which the cross-sectional sample 41 for a transmission electron microscope is disposed, an ion gun 10 used as ion gun means for emitting an ion beam 11 onto the sample 41 disposed in the working chamber 60, an electron gun 20 used as electron gun means for irradiating the worked part of the sample 41 with an electron beam 21 at an angle of approximately 90 degrees with respect to the ion beam 11 emitted by the ion gun 10, a transmission electron detector 30 used as detection means which are disposed facing the above-mentioned electron gun 20, and which receive the electron beam that

passes through the above-mentioned cross-sectional sample 41 for a transmission electron microscope, and detect the amount of current of the transmitted electron beam, and a low-voltage electrode 50 used as electrode means which are disposed so as to surround the above-mentioned sample 41 in the vicinity of the position where this sample 41 is fixed, and which absorb the secondary electrons 12 that are generated by the ion beam 11 and electron beam 21, and thus prevent the occurrence of a situation in which the accurate amount of current of the transmitted beam cannot be measured.

[0040] [The system] is constructed so that the sample 41 is conveyed into the interior of the working chamber 60 by a sample introduction system (not shown in the figures), and is appropriately driven by a stage driving system (not shown in the figures). The ion beam 11 and electron beam 12 are respectively capable of raster scanning, and [the system is] constructed so that observation of the shapes of the respective beam irradiation regions can be accomplished by a secondary ion detector or secondary electron detector (not shown in the figures). Furthermore, the operation of the present embodiment is the same as that of the embodiment of the method for producing a cross-sectional sample for a transmission electron microscope that will be described later; accordingly, a description of this operation is omitted.

[0041] Next, embodiments of the methods of Claims 3 and 4 for producing a cross-sectional sample for a transmission electron microscope will be described with reference to Figures 2a through 2g and Figures 3a through 3h. The object of the present embodiments is to provide a method for producing a cross-sectional sample for a transmission electron microscope which can automatically detect the optimal sample thickness by quantitatively monitoring the thickness of the worked surface of the transmission electron microscope sample, and which can easily judge the uniformity of the thickness of the worked part during working.

[0042] As is shown in Figure 2a, marking 43 is performed by forming holes by means of the focused ion beam apparatus or a laser marker that is equipped with a microscope, etc., around a specified microscopic part 42 for which cross-sectional observation/analysis by means of a transmission electron microscope is desired (such as a defective transistor on an LSI chip 41). Furthermore, it is advisable that this marking be performed in a position that is separated from the specified microscopic part 42 by a distance of approximately 20  $\mu\text{m}$  or greater in order to prevent the specified microscopic part 42 from being subjected to the thermal effects of the laser or ion beam irradiation used for marking 43 or contamination by flying debris from the formation of the holes. From the standpoint of positional confirmation in subsequent working, it is desirable that the size and depth of the marking be as large as possible; on the other hand, from the standpoint of the need to suppress heat and flying debris during marking, it is advisable that

the size [of the marking] be approximately 5  $\mu\text{m}$  or less, and that the depth [of the marking] be approximately 1 to 5  $\mu\text{m}$ . Using the marking as a reference, the area around the specified microscopic region for which observation or analysis is desired is cut to a size of approximately 1.5 mm square or less, which allows introduction into the transmission electron microscope, by means of the high-speed outer circumferential rotary blade 61 of a dicing machine.

[0043] In this case, as is shown in Figure 2b, surfaces that are parallel to the sectional surface of the transmission electron microscope sample 41 for which observation or analysis is desired are selected as the cut surfaces. In regard to the cutting width in the direction perpendicular to the sectional surface of the sample 41 for which observation/analysis is desired, a narrower cutting width allows a reduction in the subsequent focused ion beam working range; accordingly, cutting is performed so that the cutting width in the perpendicular direction is narrow within a range that causes no destruction such as chipping of the specified microscopic part 42 (for which observation/analysis is desired) during cutting, e.g., a width of 100 to 200  $\mu\text{m}$ . If necessary, the area in the vicinity of the surface of the part of the sample for which observation/analysis is desired is ground even thinner by the high-speed outer circumferential rotary blade 61 of the dicing machine as shown in Figure 2d.

[0044] The worked LSI chip is introduced into the focused ion beam apparatus. When the LSI chip is introduced into the focused ion beam apparatus, the orientation of the LSI chip is set so that the worked sectional surface faces the electron gun 20 inside the focused ion beam apparatus. The rectangular regions 81 and 82 which have the sectional surface for which observation/analysis is desired as one side are irradiated with the focused ion beam 11 in a raster scan by means of the focused ion beam apparatus, thus performing thin film working of the sectional surface for which observation/analysis is desired. The rectangular region 81 faces the transmission electron detector 30, and the rectangular region 82 is a region that includes a sectional surface facing the electron gun 20 inside the focused ion beam apparatus. In this focused ion beam working, the working of the region 81 is performed first. In the working of the region 81, the positional precision of the working of the sectional surface and the uniformity of the worked surface are increased while lowering the beam current/beam diameter of the focused ion beam 11 in steps.

[0045] The general working conditions are as follows: specifically, working is performed to a position that is separated from the specified microscopic part 42 constituting the target by a distance of a few microns using a Ga ion beam at an acceleration voltage of 25 to 30 kV and a beam current of approximately 2000 pA; then, working is performed to a position that is separated from the specified microscopic part 42 by a distance of 1  $\mu\text{m}$  at a beam current of

approximately 400 pA. Furthermore, working is then performed to a position that includes the specified microscopic part 42 at a beam current of approximately 100 pA, and finally, the finishing of the worked surface is performed using a beam that has a beam current of a few tens of picoamperes. Furthermore, the focused ion beam 11 has an inverted circular conical shape, so that if beam irradiation is performed perpendicular to the sample surface, a vertical sectional surface cannot be obtained; accordingly, working is performed with the sample 41 inclined at an angle of approximately 3 to 5 degrees in accordance with the beam conditions.

[0046] Following the completion of the working of the region 81, the working of the region 82 is performed by the same method. In the working of the region 82, irradiation with the electron beam 21 is performed substantially perpendicular to the sectional surface for which observation/analysis is desired in the stage where the thickness of the worked part has reached approximately 1  $\mu\text{m}$ , and the transmission electrons passing through the worked part of the sectional surface of the sample are detected by the transmission electron detector 30.

[0047] The acceleration voltage of the electron beam is set at 10 kV or greater. In the case of silicon, the electron beam will pass through a thickness of 1  $\mu\text{m}$  if the acceleration voltage is 10 kV or greater. Accordingly, as a result of this electron beam irradiation, the transmission electrons are detected by the detector 30. Furthermore, a Channeltron, etc., which has a high sensitivity and a rapid detection rate is effective as the detector 30. However, depending on the material of the sample and the setting of the electron beam current, a Faraday cup, etc., may also be used. The voltage that is applied to the detector 30 and the current of the electron beam are appropriately set in accordance with the transmission electron beam current that is detected. The electron beam can be manipulated upward and downward and to the left and right within the range of the same material as shown in Figures 3a and 3b, and when the transmission beam current is detected during this period, a uniform waveform will be obtained as shown in Figure 3c in cases where the thickness of the worked part is uniform. On the other hand, in cases where the thickness of the worked part is non-uniform as shown in Figures 3e, 3f and 3g, the transmission beam current waveform will be a waveform such as that shown in Figure 3d. The non-uniformity of the worked part confirmed in this stage can be finally corrected by correcting the focused ion beam shape and sample angle, etc., in subsequent working.

[0048] The working of the region 82 is performed by focused ion beam working while continuing the detection of the transmission beam current. As the thickness of the worked part becomes smaller, the transmission beam current increases. If the acceleration voltage of the electron beam 21 is lowered in steps in accordance with the increase in the detected transmission beam current, then the thickness through which the electron beam is transmitted also drops as

shown in Figure 3h. Accordingly, if the acceleration voltage is appropriately selected, the variation in the thickness of the worked part can be accurately detected in accordance with the variation in the transmission beam current. In the case of a silicon material, if the acceleration voltage of the final electron beam 21 is set at approximately 3 kV or less, thicknesses of approximately 500 to 1000 Å can be detected by the value of the transmission beam current. If the conditions of the amount of the transmission current are determined using a good transmission electron microscope sample, and the transmission beam current amount taken as the working endpoint is determined in advance, then the working endpoint can be automatically detected.

[0049] Furthermore, in this detection of the transmission electron beam, a deterioration in the precision of detection of the transmission electron beam current can be prevented by applying a low positive potential to the low-voltage electrode 50, and recovering large quantities of second electrons 12 generated by the irradiation with the focused ion beam. Thus, detection of the transmission electron beam current can be accomplished even during focused ion beam irradiation, so that excessive working can be prevented.

[0050] Furthermore, in order to prevent deleterious effects on the ion beam and electron beam, magnetization is prevented by using a nonmagnetic metal as the material of the low-voltage electrode 50. The voltage applied to the low-voltage electrode 50 is set at + several tens of volts so that this voltage will have no effect on the track of the focused ion beam or electron beam with [an acceleration voltage of] a few kilovolts to approximately 30 kV, and so that the recovery rate of the secondary electrons (with a voltage of several tens of electron-volts) generated by the focused ion beam irradiation is increased.

[0051] The reason that the region 81 is worked first is as follows: specifically, under conditions in which the working region 81 on the side of the sectional surface facing the transmission electron detector 30 is irradiated with the focused ion beam 11, scattered ions of the focused ion beam enter the side of the transmission electron detector 30, so that accurate measurement of the transmission beam current value becomes difficult. Accordingly, thickness evaluation and endpoint detection for the worked part by the detection of the transmission beam current amount in the stage of the working of the region 82, in which scattered ions of the focused ion beam tend not to enter the detector 30 are performed after first completing [the working of] the working region of the sectional surface on the side of the transmission electron detector 30.

[0052] As is shown in Figure 2g, [the sample 41] is pasted to the transmission electron microscope mesh 80 with the portion of the sample 41 for which analysis/observation is desired at the center. Then, observation and analysis of the sample 41 are performed by means of the

transmission electron microscope. The above has been a description of working by means of a focused ion beam; however, in regard to the evaluation of the film thickness of the worked part and uniformity of the film thickness, [this technique] can also be applied to working such as ion milling.

[0053]

[Effect of the Invention] In the focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope according to Claim 1, and the methods for producing a cross-sectional sample for a transmission electron microscope according to Claims 3 and 4, a sectional surface for observation which has a uniform thickness can be formed, and excessive working can be prevented. As a result, detection can be performed with a precision in which the variation in the thickness of the worked part is 50% [*sic*] or less, and this can be corrected in the working stage. Accordingly, the variation in the thickness of the worked part can be reduced to 50 nm or less in the final stage, so that high-resolution observations can be performed throughout more or less the entire region within the worked area. Since the sample thickness can be detected as a numerical value in the preparation of cross-sectional samples, transmission electron microscope samples with an optimal thickness can be prepared regardless of the degree of experience of the operator if the conditions are determined for each material.

[0054] In the focused ion beam apparatus for producing a cross-sectional sample for a transmission electron microscope according to Claim 2, it is possible to prevent the deterioration of the precision with which the transmission electron beam current is detected as a result of the detection by the transmission electron detector of secondary electrons generated by the ion beam and electron beam irradiation. Furthermore, detection of the transmission electron beam current is possible even during focused ion beam irradiation, so that excessive working can be prevented even during focused ion beam irradiation. As a result, the working precision of the cross-sectional sample can be improved, and the preparation of cross-sectional samples can easily be performed.

[Brief Description of the Drawings]

[Figure 1] Figure 1 is a schematic structural diagram which shows an embodiment of the focused ion beam apparatus of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2a] Figure 2a is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2b] Figure 2b is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2c] Figure 2c is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2d] Figure 2d is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2e] Figure 2e is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2f] Figure 2f is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 2g] Figure 2g is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3a] Figure 3a is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3b] Figure 3b is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3c] Figure 3c is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3d] Figure 3d is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3e] Figure 3e is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3f] Figure 3f is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3g] Figure 3g is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 3h] Figure 3h is a diagram which illustrates an embodiment of the method of the present invention for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4a] Figure 4a is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4b] Figure 4b is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4c] Figure 4c is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4d] Figure 4d is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4e] Figure 4e is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4f] Figure 4f is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 4g] Figure 4g is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5a] Figure 5a is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5b] Figure 5b is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5c] Figure 5c is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5d] Figure 5d is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5e] Figure 5e is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

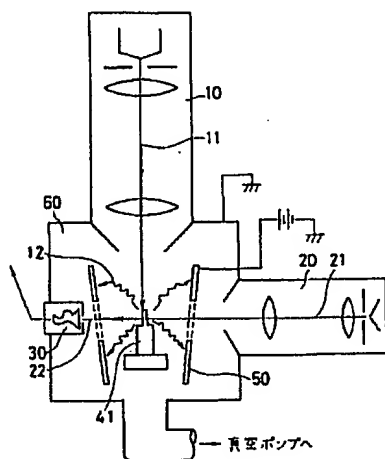
[Figure 5f] Figure 5f is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 5g] Figure 5g is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

[Figure 6] Figure 6 is a diagram which illustrates a conventional method for producing a cross-sectional sample for a transmission electron microscope.

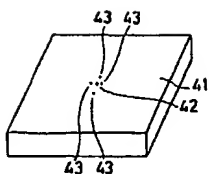
[Explanation of Symbols]

- 10: Ion gun
- 20: Electron gun
- 30: Transmission electron detector
- 40 [sic]<sup>†</sup>: Sample
- 50: Low-voltage electrode

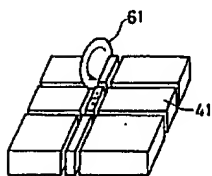


[Figure 1]

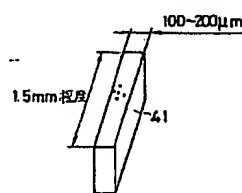
[Key:] To vacuum pump



[Figure 2a]

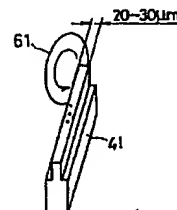


[Figure 2b]



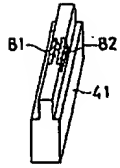
[Figure 2c]

[Key:] Approximately 1.5 mm

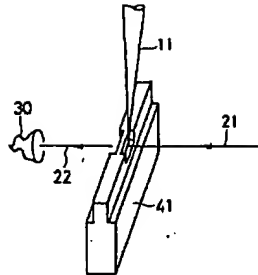


[Figure 2d]

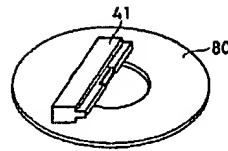
<sup>†</sup> Translator's note: apparent error in the original for "41."



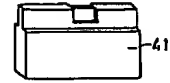
[Figure 2e]



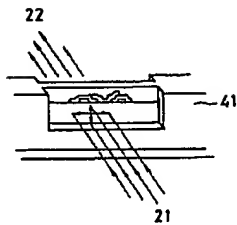
[Figure 2f]



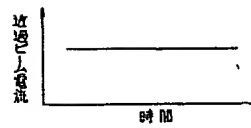
[Figure 2g]



[Figure 3a]

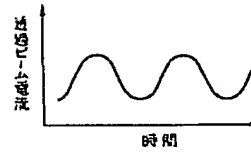


[Figure 3b]



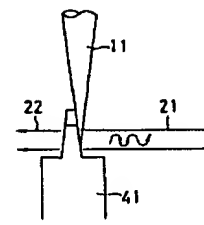
[Figure 3c]

[Y axis:] Transmission beam current  
[X axis:] Time

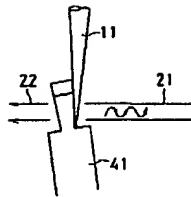


[Figure 3d]

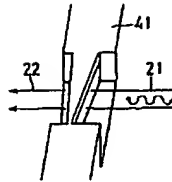
[Y axis:] Transmission beam current  
[X axis:] Time



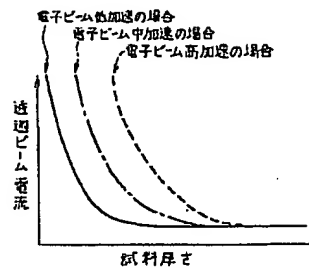
[Figure 3e]



[Figure 3f]



[Figure 3g]



[Figure 3h]

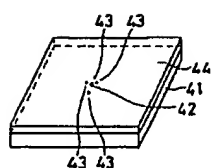
[Y axis:] Transmission beam current

[X axis:] Sample thickness

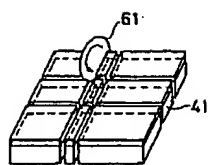
[In graph, left:] In the case of low acceleration of the electron beam

[In graph, middle:] In the case of medium acceleration of the electron beam

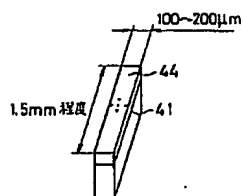
[In graph, right:] In the case of high acceleration of the electron beam



[Figure 4a]

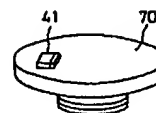


[Figure 4b]

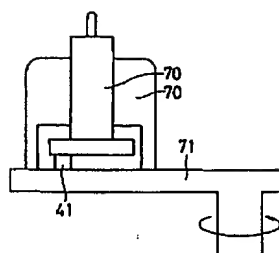


[Figure 4c]

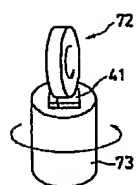
[Key:] Approximately 1.5 mm



[Figure 4d]



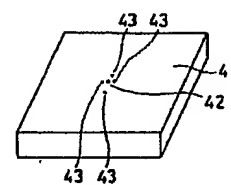
[Figure 4e]



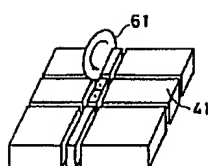
[Figure 4f]



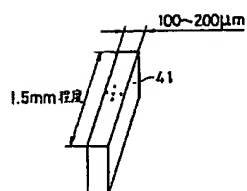
[Figure 4g]



[Figure 5a]

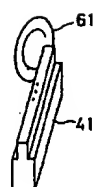


[Figure 5b]

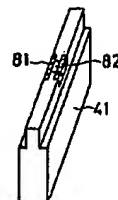


[Figure 5c]

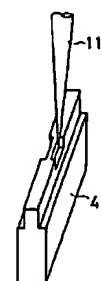
[Key:] Approximately 1.5 mm



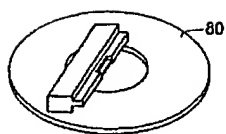
[Figure 5d]



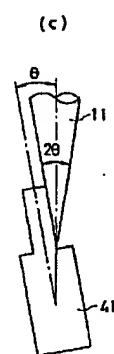
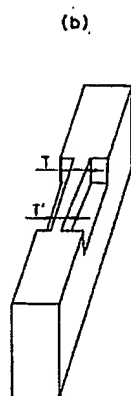
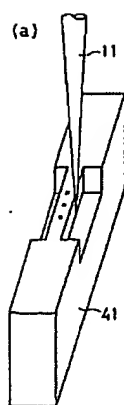
[Figure 5e]



[Figure 5f]



[Figure 5g]



[Figure 6]

Continued from the front page

(51) Int. Cl.<sup>6</sup>  
H 01 J 37/31

Identification Symbol

JPO File No. F1  
9172-5E

Technical Indication